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Indian Standard

IS 17342 : 2020

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Determination of Ethyl Alcohol Concentration by Digital Densitometer

ICS 71.040.10

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भारतीय मानक ब्यूरो
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Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

There are diverse methods for determining the ethanol content of alcoholic liquids and their preparations. These methods are based on the density, refractive index, oxidation index, gas chromatographic retention time, etc. The simplest and the most widely used method is the one based on the density of ethanol-water mixtures, which decrease with the increase of ethanol content in a manner which has been determined to the highest degree of accuracy.

The density data on alcohol-water mixtures as a function of temperature made available by Organisation Internationale de Metrologie Legale/International Organization of Legal Metrology (OMIL) is the ultimate accuracy available to date and is a collaborative work based on calculations (by computer) on the results of measurements made by U. S. National Bureau of Standards, National Bureau of Quality and Measures, Poland, National Research Laboratory of Metrology, Japan, Montepellier Faculty of Pharmacy, France, Physikalisch Techniche Bundesanstalt, F.R. Germany, and the International Union of Pure and Applied Chemistry.

The OIML data in so far as ethanol content by volume is concerned, uses 20°C as reference temperature, that is, real strength by volume defined as volume of ethanol at 20°C in 100 volumes of the alcoholic sample at 20°C.

Computation of alcohol strength of aqueous-alcoholic solutions could be done by pyknometry, hydrometery or oscillating sample tube method (digital densitometer). For digital densitometry the direct usable table of apparent specific gravity t°/t° in the range of Indian ambient temperatures 10 to 40°C based on OIML data are available in IS 3506.

In the preparation of this standard considerable assistance has been derived from following standards:

IS 15464 : 2004	Anhydrous ethanol for use in automotive fuel — Specification
AOAC 982.10 : 2016	Alcohol by Volume in distilled Liquors, Densitometric Method
OIV- 8/2000-377/2009	Alcoholic strength by volume-Type I methods

The composition of the Committee, responsible for the formulation of this standard is given at Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 :1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

DETERMINATION OF ETHYL ALCOHOL CONCENTRATION BY DIGITAL DENSITOMETER

1 SCOPE

This standard prescribes the method for the determination of ethyl alcohol in anhydrous ethanol and aqueous alcoholic solution, by using a digital densitometer.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute the provisions of the standards. At the time of publication, the editions indicated are valid. All standards are subject to revision, and parties to investigate the possibility of applying the most recent editions of standard indicated below:

IS No.	*Title*
2302 : 1989	Tables for alcoholometry by hydrometer method (*first revision*)
3506 : 1989	Tables for alcoholometry (Pyknometer method) (*first revision*)
1070 : 1992	Reagent grade water (*third revision*)

3 TERMINOLOGY

For the purposes of this standard, besides the definitions given in IS 2302 and the following shall apply.

3.1 Ethanol — Ethanol is the pure chemical, otherwise known as hydroxyl ethane, corresponding to the constitution $CH_3.CH_2OH$ and molecular formula C_2H_5OH.

3.2 Anhydrous Ethanol — Anhydrous ethanol is essentially ethyl alcohol, which is denatured and is meant for use as fuel in automobile engines.

3.3 Standard Atmospheric Conditions — Standard atmospheric conditions for testing shall comprise a relative humidity of 65 ± 2 percent and a temperature of 27 ± 2 °C provided that, in a given series of experiments, the temperature does not vary by more than ± 1 °C (*see* IS 196).

3.4 Density — The density of a sample at a given temperature is the physical property of the sample which equals to the ratio of its mass to the volume. Mass is independent of external conditions, such as buoyancy in air or gravity. It corresponds to weight in vacuum.

$$d = m / V, \text{ expressed as g / ml}$$

Where,

d = Density of the liquid;
m = Mass; and
V = Volume.

3.5 Specific Gravity — It is the ratio of measured density of a sample and the density of water at a certain temperature.

$$SG = d_{\text{sample}} / d_{\text{water}}$$

Where,

d_{sample} = density of the sample at specific temperature; and
d_{water} = density of the water at specific temperature.

3.6 Apparent Relative Density (Apparent Specific Gravity) — It is the relative density/specific gravity in air obtained by the pyknometer method or densitometer at t °C with respect to water at t°C.

$$SGa_{pp}\,20/20 = \frac{d_{\text{app sample}}@\,20°C}{d_{\text{app water}}@\,20°C}$$

Where,

$d_{\text{app sample}}$@ 20°C = apparent density of sample at 20°C; and
$d_{\text{app water}}$@ 20°C = apparent density of water at 20°C.

3.7 Single Bulk Method — When a measured volume, say 100 ml, of a spirituous preparation is diluted and distilled to collect the same volume of distillate, that is 100 ml, it is referred as single bulk method.

3.8 Double Bulk Method — When a measured volume, say 50 ml, of a spirituous preparation is diluted and distilled to collect distillate twice the original volume of the sample, that is 100 ml, it is referred as the double bulk method.

3.9 Treble Bulk Method — When a measured volume, say 25 ml, of a spirituous preparation is diluted and distilled to collect thrice the volume of the sample, that is 75 ml, it is referred to as treble bulk method.

3.10 Quadruple Bulk Method — When a measured volume, say 25 ml, of a spirituous preparation is diluted to collect four times the volume of the sample, that is 100 ml, it is referred to as quadruple bulk method.

4 DETERMINATION OF THE ETHANOL CONCENTRATION OF A SPIRITUOUS SOLUTION

4.0 General

Ethanol content of a spirituous preparation can be quantitatively determined by specific gravity determination. However, prior to this determination, ethanol contained in the preparation as a rule, has to be obtained practically free from all other dissolved and undissolved substances except water. Simple direct distillation suffices where the admixed or dissolved ingredients are not volatile with steam. When volatile bodies are present, it is necessary either to render them incapable of distillation or to remove them. All spirituous preparations containing volatile acids or ammonia (or amines) are neutralized by an alkali or acid (sodium hydroxide or sulphuric acid). Free iodine, if present, may first be converted into sodium iodide by treatment with sodium thiosulphate. Volatile oils, solvents, etc, are removed by adopting the single, double, treble or quadruple bulk method as described in **3.3** of IS 3506. A definite volume of distillate is collected and the specific gravity determined by pyknometer method or electronic densitometer. Through suitable use of Table 1 and Table 2 (IS 3506), ethanol content of sample determined.

4.1 Principle

The liquid density is determined by electronic measurement of the oscillations of vibrating U-tube. The principle of method consists firstly of distilling the sample (if it is containing other than water constituents) volume by volume. The distillation procedure is described in IS 3506. This distillation enables the elimination of non-volatile substances. The distillate density of the distillate is measured.

4.1.1 A small volume (approximately 0.7 ml) of liquid sample is introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data of instrument to determine the density of the sample.

4.2 Apparatus

4.2.1 *Distillation Assembly* — Please refer IS 3506.

4.2.2 *Digital Density Analyzer* — Consisting of a U-shaped, oscillating sample tube and a system for electronic excitation, frequency counting, and display. The analyzer must accommodate the accurate measurement of the sample temperature during measurement or must control the sample temperature as described in **4.2.3**. The instrument shall be capable of expressing the density in g /ml to 5 decimal places.

4.2.3 *Circulating Constant-Temperature Bath* — Optional capable of maintaining the temperature of the circulating liquid constant to ± 0.05°C in the desired range. Temperature control can be maintained as a part of the density analyzer instrument package.

4.2.4 *Sample Injection Syringes* — At least 2 ml in volume with a tip or an adapter tip that will fit the opening of the oscillating tube.

4.2.5 *Autosampler* — Require for the use in automated injection analyses. The auto sampler shall be designed to ensure the integrity of the sample prior to and during the analysis and be quipped to transfer a representative portion of sample to the digital density meter.

4.2.6 *Flow -Through or Pressure Adapter* — For use as an alternative means of introducing the sample into the density analyzer either by pump or by vacuum.

4.2.7 *Thermometer* — Calibrated and graduated to 0.1°C, and a thermometer holder that can be attached to an instrument for setting and observing the test temperature. In calibrating the thermometer, the ice point and bore connections should be estimated to the nearest 0.05°C.

4.3 Reagents and Materials

4.3.1 *Double Distilled Water or of an Equivalent Analytical Purity (see IS 1070)*

4.3.2 *Acetone,* for flushing and drying the sample tube.

NOTE — **Warning**: Extremely flammable.

4.3.3 *Petroleum Naphtha*, for flushing viscous petroleum samples from the sample tube.

NOTE — **Warning**: Extremely flammable.

4.3.4 *Dry Air,* for blowing the oscillator tube.

4.3.5 *Undenatured Concentrated Ethanol,* as a solvent

4.4 Preparation of Apparatus

Set up the density analyzer and constant temperature bath following the manufacturer's instructions. Adjust the bath or internal temperature control so that the desired test temperature is established and maintained in the sample compartment of the analyzer. Calibrate the instrument at the same temperature at which the density of the sample is to be measured.

NOTE — **Caution**: Precise setting and control of the test temperature in the sample tube is extremely important. An error of 0.1°C can result in a change in density of one in the fourth decimal.

4.5 Calibration of Apparatus

4.5.1 Calibrate the instrument when first set up and whenever the test temperature is changed. Thereafter, conduct calibration checks at weekly intervals during routine operation.

4.5.2 Initial calibration, or calibration after a change in test temperature, necessitates calculation of the values of the constants A and B from the periods of oscillation (*T*) observed when the sample cell contains air and redistilled, freshly boiled and cooled reagent water. Other calibrating materials, such as *n*-nonane, *n*-tridecane, cyclohexane, and *n*-hexadecane (for high temperature applications) can also be used as appropriate.

4.5.2.1 While monitoring the oscillation period, *T*, flush the sample tube with petroleum naphtha, followed with an acetone flush and dry with dry air. Contaminated or humid air can affect the calibration. When these conditions exist in the laboratory, pass the air used for calibration through a suitable purification and drying train. In addition, the inlet and outlet ports for the U-tube must be lugged during measurement of the calibration air to prevent ingress of moist air.

4.5.2.2 Allow the dry air in the U-tube to come to thermal equilibrium with the test temperature and record the T-value for air.

4.5.2.3 Introduce a small volume (about 0.7 ml) of redistilled, freshly boiled and cooled reagent water into the sample tube from the bottom opening using a suitable syringe. The test portion must be homogeneous and free of even the smallest air or gas bubbles. The sample tube does not have to be completely full as long as the liquid meniscus is beyond the suspension point. Allow the display to reach a steady reading and record the *T*-value for water.

4.5.2.4 Calculate the density of air at the temperature of rest using the following equation:

$$d_m, \text{g/ml} = 0.001\,293\ [273.15/T]\ .\ [P/760] \qquad \ldots(1)$$

Where,

T = Temperature, K, and

P = Barometric pressure, torr.

4.5.2.5 Determine the density of water at the temperature of test by reference to Table A.

4.5.2.6 Using the observed T-values and the reference values for water and air, calculate the values of the constants A and B using the following equations:

$$\text{A} = [T_w^{\,2} - T_a^{\,2}] \,/\, [d_w - d_a] \qquad \ldots(2)$$

$$\text{B} = T_a^{\,2} - (A \times d_a) \qquad \ldots(3)$$

Where,

T_w = Observed period of oscillation for cell containing water,

T_a = Observed period of oscillation for cell containing air,

d_w = Density of water at test temperature, and

d_a = Density of air at test temperature.

Alternatively, use T and d values for the other reference liquid, if one is used.

4.5.2.7 If the instrument is equipped to calculate density from the constants A and B and the observed T-values from the sample, then enter the constants in the instrument memory in accordance with the manufacturer's instructions.

4.5.2.8 Check the calibration and adjust, if needed by performing the routine calibration check described in **4.5.3.2**.

4.5.2.9 To calibrate the instrument to display relative density, that is, the density of the sample at a given temperature referred to the density of water at the same temperature, follow sections **4.5.2.1** through **4.5.2.7**, but substitute 1.000 for d_w in performing the calculations described in **4.5.2.6.**

4.5.3 Weekly calibration adjustments to constants, A and B can be made if required, without repeating the calculation procedure.

NOTE — The need for a change in the calibration is generally attributable to deposits in the sample tube that are not removed by the routine flushing procedure. Although this condition can be compensated by adjusting A and B, it is good practice to clean the tube with warm chromic acid solution.

Warning — Causes severe burns. A recognized carcinogen whenever a major adjustment is required. Chromic acid solution is the most effective cleaning agent, however, surfactant cleaning fluids have also been used successfully.

4.5.3.1 Flush and dry the sample tube as described in **4.5.2.1** and allow the display to reach a steady reading. If the display does not exhibit the correct density for air at the temperature of rest, repeat the cleaning procedure or adjust the value of constant B commencing with the last decimal place until the correct density is displayed.

4.5.3.2 If adjustment to constant B was necessary in **4.5.3.1** then continue the recalibration by introducing redistilled, freshly boiled and cooled reagent water into the sample tube as described in **4.5.2.3** and allow the display to reach a steady reading. If the instrument has been calibrated to display the density, adjust the reading to the correct value for water at the test temperature (*see* Table A) by changing the value of constant A, commencing with the last decimal place.

If the instrument has been calibrated to display the relative density, adjust the reading to the value 1.000 0.

NOTE — In applying this weekly calibration procedure, it can be found that more than one value each for A and B, differing in the fourth decimal place, will yield the correct density reading for the density of air and water. The setting chosen would then be dependent upon whether it was approached from a higher or lower value. The setting selected by this method could have the effect of altering the fourth place of the reading obtained for a sample.

4.5.4 Some analyzer models are designed to display he measured period of oscillation only (*T*-values) and their calibration requires the determination of an instrument constant K, which must be used to calculate the density or relative density from the observed data.

4.5.4.1 Flush and dry the sample tube as described in **4.5.2.1** and allow the display to reach a steady reading. Record the *T*-value for air.

4.5.4.2 Introduce redistilled, freshly boiled and cooled reagent water into the sample tube as described in **4.5.2.3,** allow the display to reach a steady reading and record the *T*-value for water.

4.5.4.3 Using the observed T-values and the reference values for water and air (**4.5.2.4** and **4.5.2.5**), calculate the instrument constant K using the following equations:

$$K = [1.000\,0 - d_a] / [T_w^{\,2} - T_w^{\,2}] \qquad \ldots(4)$$

Where,

T_w = Observed period of oscillation for cell containing water,

T_a = Observed period of oscillation for cell containing air, and

d_a = Density of air at test temperature.

4.6 Procedure

4.6.1 *Sample Preparation*

If the sample contains other admixed or dissolved ingredients that are not volatile with steam, a simple direct distillation procedure needs to be performed on the sample before analysis. If other volatile bodies are present, it is necessary either to render them incapable of distillation or to remove them**.** For distillation please refer **3.3** of IS 3506

4.6.2 *Manual Injection*

4.6.2.1 Introduce a volume of about 0.7 ml of sample into a clean and dry U-tube of the instrument using a suitable syringe.

4.6.2.2 The sample can also be introduced by siphoning. Plug an external TFE-fluorocarbon or silicone capillary tube into the lower entry port of the sample tube. Immerse the other end of the capillary tube in the sample and apply suction to the upper entry port using a syringe or vacuum line until the sample tube is properly filled.

4.6.3 *Automatic Injection*

The use of an auto sampler (*see* **4.2.5**) is required when analyzing samples by automated injection. Follow the manufacturer's instructions for ensuring the integrity of the sample prior to analysis, as well as transferring a representative sample into the instrument for analysis.

4.6.4 Turn on the illumination light and examine the sample tube carefully. Make sure that no bubbles are trapped in the tube, and that it is filled to just beyond the suspension point on the right-hand side. The sample must be homogenous and free of even the smallest gas bubbles.

NOTE — Digital Densitometers are equipped with optical or physical bubble detection system.

4.6.5 Turn the illumination light off immediately after sample introduction, because the heat generated can affect the measurement temperature.

4.6.6 After the instrument displays a steady reading to four significant figures for density and five for *T*-values, indicating that temperature equilibrium has been reached, record the density or *T*-values

4.7 Calculation

4.7.1 *Calculating Density Analyzers*

The recorded is the final result, as relative density.

4.7.2 *Non-calculating Density Analyzers*

Using the observed *T*-value for the sample and the *T*-value for water and appropriate instrument constant, *K,* determine in **4.5.4.3,** calculate the density or relative density using equation 5. Carry out all calculations to six significant figures and round the final results to four.

$$\text{Relative density, t/t} = 1 + K\,(T_s^{\,2} - T_w^{\,2}) \qquad \ldots(5)$$

Where,

T_w = Observed period of oscillation for cell containing water,

T_s = Observed period of oscillation for cell containing sample,

K = Instrument constant, and

t = Temperature of test, °C.

4.7.3 *Calculation for Alcoholic Strength v/v Percent by Volume*

4.7.3.1 Electronic densitometers usually have an inbuilt system for applying the alcoholometry tables (depending on the country/ industry requirement). The application of the tables directly gives the alcoholic strength by the volume.

4.7.3.2 If the electronic densitometer is not producing the direct alcoholic strength of the liquid, the alcoholic strength by volume is derived by using OIML Table 1 and 2 of IS 3506 .

4.7.3.3 *Table 1*

Gives the specific gravity or relative density in air at various temperatures (10 to 40°C at intervals of 1°C) for various percentage of ethanol (by volume at 20°C). With the help of this table the measured specific gravity in air of an alcoholic liquid at a particular temperature, can be converted into its corresponding real strength that is, ethanol content by volume at 20°C.

4.7.3.4 *Table 2*

Gives the minor corrections required for converting the results of real strength, that is, ethanol content by volume at 20°C, to that at 15°C or 15.6°C as may be required for regulatory purpose until the international system of ethanol content at 20°C is adopted for local governing authorities.

5 REPORT

5.1 In reporting density or density related quantities, give the test temperature and the units, for example, density at 20°C = … g/mL (in vacuo).

5.2 In reporting relative density give both the test temperature and the reference temperature but no units. For example, relative density of sample at 27°C/27°C = density of sample at 27°C / density of water at 27°C (in vacuao).

5.3 In reporting alcoholic strength by volume, @ 20°C in percent v/v.

5.3.1 *Example*

For an alcoholic solution at 20.0°C the determined relative density (through densitometer) = 0.949 71.

Alcoholmetry table-1 (*Refer* **4.7.3.3**) derived value for sample concentration at 20.0°C =40.0 percent v/v.

5.4 Report the final density result to five significant figures.

Table A Density of Water

(*Clauses* 4.5.2.5 *and* 4.5.3.2)

Temperature °c (1)	**Density** g/ml (2)
0.0	0.999 040
3.0	0.999 964
4.0	0.999 972
5.0	0.999 964
10.0	0.999 099
15.0	0.999 099
15.56	0.999 012
16.0	0.990 943
17.0	0.990 774
18.0	0.998 595
19.0	0.990 404
20.0	0.998 203
21.0	0.997 991
22.0	0.997 709
23.0	0.997 537
24.0	0.997 295
25.0	0.997 043
26.0	0.996 702
27.0	0.996 511
28.0	0.996 231
29.0	0.995 943
30.0	0.995 045
35.0	0.994 029
37.7	0.993 042
40.0	0.992 212
45.0	0.990 200
50.0	0.900 030
55.0	0.905 600
60.0	0.903 191
65.0	0.900 546
70.0	0.977 759
75.0	0.974 837
80.0	0.971 705
85.0	0.900 606
90.0	0.965 305
100	0.950 345

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Organic, Chemicals Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative*
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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of ‘BIS Catalogue’ and ‘Standards: Monthly Additions’.

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